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IS 15623 (2005): Melamine (2 , 4, 6 triamino 1, 3, 5 triazine) [PCD 12: Plastics]

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भारतीय मानक

मेलेमाइन (2, 4, 6 ट्राईअमीनो 1, 3, 5 ट्राईएज़ीन) — विशिष्टि

Indian Standard

MELAMINE (2, 4, 6 TRIAMINO 1, 3, 5 TRIAZINE) — SPECIFICATION

ICS 71.080.90

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 2005

Price Group 3

Plastics Sectional Committee, PCD 12

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Melamine (2, 4, 6 Triamino 1, 3, 5 Triazine) is a base chemical, available in fine powder form and used to prepare melamine formaldehyde resin of different M: F ratio depending upon the application of resin. The melamine formaldehyde resin so produced is further modified that is either methoxylated or butylated to get different variety of resin to suit its application.

The melamine formaldehyde resin or derivative so produced finds application as reactive component during the manufacture of paint, adhesive, lacquer, textile treatment chemical, and super-plasticizer as construction chemicals in cement industries.

Conversion of melamine formaldehyde resin into moulding powder, and use of melamine formaldehyde resin in decorative laminate, plywood sheet, medium density particle board, high density particle board and veneer board is well known.

Recent development shows that melamine and its derivatives like melamine polyphosphate, melamine-cyanurate with additives find applications as flame retardant for plastics and polymers and also as fire extinguishing dry chemical powder due to its high heat capacity and decomposition at very high temperature.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MELAMINE (2, 4, 6 TRIAMINO 1, 3, 5 TRIAZINE) — SPECIFICATION

1 SCOPE

This standard covers the requirements and methods of sampling and test for melamine.

2 REFERENCES

The following standards contain provisions, which through reference in the text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
265 : 1993	Hydrochloric acid (*fourth revision*)
1070 : 1992	Reagent grade water (*third revision*)
4905 : 1968	Methods for random sampling
8768 : 2000	Method of measurement of colour in liquid chemical products platinum-cobalt scale (*second revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a white crystalline powder and free from foreign impurities.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods given in col 4 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable packing media (multi-wall craft paper bag) or containers as agreed to between the purchaser and the supplier.

4.2 Marking

The container shall be marked with the following information:

- a) Name of the material;
- b) Manufacturer's name, initials or trade-mark, if any;
- c) Date/month of manufacture;
- d) Net mass of material; and
- e) Identification in code or otherwise to enable the lot of consignment or manufacture to be traced back from records.

4.2.1 *BIS Certification Marking*

The container may also be marked with the Standard Mark.

4.2.1.1 The use of Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING AND CRITERIA FOR CONFORMITY

Representative samples of the material shall be drawn and criteria for conformity of the material to the requirement of this specification shall be determined according to the procedure prescribed in Annex H.

6 TESTS

6.1 Tests shall be carried out as given in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, A.R. Grade chemicals and distilled water shall be used in tests.

Table 1 Requirements for Melamine (2, 4, 6 Triamino 1, 3, 5 Triazine)

(*Clause* 3.2)

Sl No.	Characteristics	Requirements	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Appearance	White crystalline powder, free from foreign impurities	A
ii)	Assay percent. *Min*	99.8	B
iii)	Loss on drying, percent. *Max*	0.1	C
iv)	Ash content, percent. *Max*	0.01	D
v)	*p*H	7.0-8.5	E
vi)	Colour, APHA, *Max*	20	F
vii)	Clarity	Clear to slight turbid	G

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF APPEARANCE

A-1 SCOPE

The method covers visual inspection of melamine.

A-2 PROCEDURE

Place approximately 50 g melamine on a clean white paper of 21 cm × 30 cm size. Visually examine and report the colour and impurities.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF ASSAY/PURITY

B-1 SCOPE

The method covers the gravimetric procedure to determine purity of melamine.

B-2 APPARATUS

B-2.1 Beaker, 500 ml.

B-2.2 Glass Rod

B-2.3 Sintered Glass Crucible G-4

B-2.4 Analytical Balance, accuracy ± 0.000 1 g.

B-2.5 Oven

B-2.6 Hot Plate

B-2.7 Desiccators

B-3 REAGENTS

B-3.1 Cyanuric Acid GR

B-3.2 Washing Liquid, 0.3 g cyanuric acid/litre.

B-4 PROCEDURE

B-4.1 Weigh accurately about 200 mg of sample and transfer it to 500-ml beaker. Add about 300 ml of distilled water and heat until sample is dissolved.

B-4.2 In another 250 ml beaker, take 300 mg of cyanuric acid, add 100 ml distilled water and warm till cyanuric acid gets dissolved. Add this hot cyanuric acid solution to melamine solution. Heat the mixture just below boiling. Cool to the room temperature. Melamine-cyanurate will get precipitated. Allow it to stand for 2 h. Filter the precipitate through previously dried and weighed G-4 sintered glass crucible and transfers the precipitate quantitatively into crucible using washing liquid. Finally wash the precipitate with minimum amount of distilled water. Take care that the total washing should not exceed 25 ml. Dry the crucible for 1 h at 105°C in hot air oven. Cool the G-4 crucible in desiccator, weigh and finally determine the weight of melamine-cyanurate.

B-5 CALCULATION

$$\text{Melamine, percent by mass} = \frac{M_2 \times 0.494\,2 \times 100}{M_1}$$

where

M_1 = mass of the sample, and

M_2 = mass of melamine-cyanurate precipitate.

NOTE — 0.494 2 is a factor for converting mass of melamine-cyanurate to mass of melamine.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF LOSS ON DRYING

C-1 SCOPE

The method covers the analytical procedure to determine moisture content by drying the sample in oven at 105 °C.

C-2 APPARATUS

C-2.1 Weighing Bottle with Stopper

C-2.2 Desiccator

C-2.3 Oven

C-2.4 Analytical Balance, accuracy ± 0.000 1 g.

C-3 PROCEDURE

Weigh accurately about 10 g sample in a weighing bottle. Dry it in the oven at 105 ± 2°C for 3 h. Cool it in desiccator and weigh it.

C-4 CALCULATION

$$\text{Loss on drying, pcercent by mass} = \frac{\text{Loss in mass} \times 100}{\text{Mass of sample}}$$

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF ASH CONTENT

D-1 SCOPE

The method covers the analytical procedure to determine non-volatile matter of melamine by ignition at 800°C.

D-2 APPARATUS

D-2.1 Silica Dish, 100 ml.

D-2.2 Spatula

D-2.3 Pair of Tongs with Platinum Tip

D-2.4 Desiccator

D-2.5 Burner

D-2.6 Analytical Balance, accuracy ± 0.000 1 g.

D-2.7 Muffle Furnace

D-3 PROCEDURE

Weigh accurately about 100 g of sample and transfer it into initially weighed silica dish. Ignite the content of the dish carefully on burner. After complete ignition, transfer the dish with content into muffle furnace having 800 ± 2°C temperature and keep it for about 1 h. Cool the dish to room temperature in desiccator. Weigh the dish on balance.

D-4 CALCULATION

$$\text{Ash content, percent by mass} = \frac{\text{Mass of residue} \times 100}{\text{Mass of sample}}$$

ANNEX E

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF *p*H (10 PERCENT SUSPENSION)

E-1 SCOPE

The method covers the analytical procedure to determine *p*H of 10 percent suspension of melamine in neutral distilled water.

E-2 APPARATUS

E-2.1 *p*H Meter with Electrode

E-2.2 Beaker, 250 ml.

E-2.3 Magnetic Stirrer

E-2.4 Top Pan Balance, accuracy ± 0.01 g.

E-2.5 Stirring Bar

E-3 REAGENTS

E-3.1 Distilled Water (*see* IS 1070).

E-3.2 N/100 HCl

E-3.3 N/100 NaOH

E-4 PROCEDURE

Take 100 ml of distilled water in 250 ml beaker. Measure the *p*H of the water. Adjust the *p*H of the water to 7.0 by either adding N/100 hydrochloric acid or sodium hydroxide. Add 10 ± 0.1g melamine into the 250 ml beaker and stir the suspension. After 3 min, check the *p*H of suspension, till constant to one decimal and report.

ANNEX F

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF COLOUR (APHA)

F-1 SCOPE

The method covers the analytical procedure to visually determine the colour (APHA) of melamine formaldehyde resin/solution by its comparison with the standard APHA solutions.

F-2 APPARATUS

F-2.1 Colorimetry Tubes, 100 ml.

F-2.2 Volumetric Flask, 250 and 500 ml.

F-2.3 Burette, 50 ml.

F-2.4 Spectrophotometer

F-3 REAGENTS

F-3.1 Potassium Hexachloroplatinate (K_2PtCl_6)

F-3.2 Cobalt Chloride Hexahydrate ($CoCl_2.6H_2O$)

F-3.3 Hydrochloric Acid (*see* IS 265)

F-3.4 Formaldehyde

F-4 PROCEDURE

F-4.1 Preparation of Solution

Transfer 50 g of melamine into 250 ml conical flask. Add 86 ml of formaldehyde solution. Heat it on hot plate to 85 ± 1°C. Stir the suspension on magnetic stirrer for about 3 min.

F-4.2 Taking 50 ml of the solution in **F-4.1** carry out the test as prescribed in IS 8768. Compare the colour of melamine formaldehyde solution with standard platinum-cobalt standards and report.

ANNEX G

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF CLARITY

G-1 SCOPE

The method covers the analytical procedure for visually determining clarity of melamine formaldehyde (MF) resin prepared by dissolving specified quantity of melamine in formaldehyde solution (Formaldehyde: Melamine 3:1 mol / mol ratio) at increased temperature, after specified period by comparison with standard turbidity solution.

G-2 APPARATUS

G-2.1 Conical Flask, 250 ml.

G-2.2 Colorimetric Tube

G-2.3 Thermometer, 0°C-110°C.

G-2.4 Hot Plate

G-2.5 Oven

G-2.6 Top Loading Balance, accuracy ± 0.01 g.

G-2.7 Measuring Cylinder

G-2.8 Volumetric Flask, 1 000 ml.

G-2.9 Magnetic Stirrer with Hot Plate

G-2.10 Pipette, 100 ml.

G-2.11 Pipette (Graduated), 10 ml.

G-2.12 Magnet Bar

G-3 REAGENTS

G-3.1 Formaldehyde, having concentration 36.0 percent, *Min.*

G-3.2 Turbidity Standard

G-3.2.1 *Hydrazine Sulphate,* 0.5 g hydrazine sulfate in 50 ml distilled water keep this solution for 5 to 6 h.

G-3.2.2 *Hexamine,* 2.5 g of hexamine in 25 ml distilled water.

G-4 PROCEDURE

G-4.1 Preparation of Standard Turbidity Solution

G-4.1.1 *Base Solution* 1

Take 25 ml of hydrazine sulfate solution into 25 ml hexamine solution the mixture is kept for 24 h. The resulting suspension is kept for 2 months.

G-4.1.2 *Base Solution* 2

From the above solution take 15 ml solution and transfer it in 1 000 ml volumetric flask. Dilute it with distilled water to the mark.

G-4.1.3 *Turbidity Standards*

Turbidity standards are as under:

a) 0 ml Base 2 solution + 100 ml distilled water: Clear

b) 5 ml Base 2 solution + 95 ml distilled water: Almost Clear

c) 10 ml Base 2 solution + 90 ml distilled water: Slight Turbid

d) 20 ml Base 2 solution + 80 ml distilled water: Turbid

NOTE — The standard solution's stability can last for about 2 weeks.

G-4.2 Resin Preparation for Turbidity Check

Transfer 50 g of melamine into 250 ml conical flask. Add 86 ml of formaldehyde solution. Heat it on hot plate to 85 ± 1°C. Stir the suspension on magnetic stirrer for about 3 min.

G-5 OBSERVATION

After additional 2 min compare turbidity of melamine formaldehyde solution with the standard turbidity solutions and report the observation.

ANNEX H

(*Clause* 5)

SAMPLING OF MELAMINE POWDER

H-1 GENERAL

In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

a) Samples shall not be taken in an exposed place.

b) The sampling instrument shall be made of stainless steel or any other suitable material on which the material shall have no action. The instrument shall be clean and dry.

c) Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

d) The sample shall be placed in suitable, clean, dry, airtight metal or glass containers on which the material has no action. The sample containers shall be of a size that the sample nearly fills the container.

e) Each sample container shall be sealed airtight with a stopper after filling and marked with full details of sampling, such as the date of sampling, the month and year of manufacture of the material, etc.

f) Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

H-2 SCALE OF SAMPLING

H-2.1 Lot

In a single consignment, all the containers of the same class, same type, same form and belonging to the same batch of manufacture, shall be grouped together to constitute a lot.If a consignment is known to consist of containers belonging to different batches of manufacture or different forms, the containers belonging to the same batch of manufacture and the same form shall be grouped together and each such group shall constitute a lot.

H-2.2 A number of containers, consisting 10 percent of the containers in a lot but not less than 3 containers in any case, shall be selected at random from a lot for the purpose of drawing samples for test (*see* IS 4905).

H-3 TEST SAMPLES AND REFERENCE SAMPLE

H-3.1 Preparation

To prepare a set of test sample, draw with an appropriate sampling instrument, from freshly opened containers which have been selected for sampling, an equal number of scoop-full of material from any point, at least 75 mm below the surface and 75 mm above the bottom of large containers, and from any point, at least 25 mm below the surface and 25 mm above the bottom of small containers. The sample prepared by mixing the portions from each container shall be not less than eight times the quantity, which is estimated to be required for carrying out all the tests. Divide this composite sample into the required number of reduced samples. Each set of these reduced samples shall constitute a lot.

H-3.2 Three sets of test samples, each not less than twice the quantity required for the purpose of testing, representative of each selected container (*see* **H-3.1**) shall be transferred immediately to thoroughly dried container, which shall be sealed airtight with an appropriate stopper. These containers shall be marked with all the particulars of sampling given under **H-1(e)**.

H-3.3 Referee Sample

The third set of the test samples, bearing the seals of the purchaser and the supplier shall constitute the referee sample, to be used in case of dispute between the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier.

H-4 TEST FOR ACCEPTANCE

H-4.1 Examination and Tests

The purchaser may examine and test separately samples from each lot (*see* **H-2.1**) for compliance with the requirements of the standard, or he may prepare for the purpose of such examination, a composite sample representing the whole of the consignment by mixing the test samples.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 2323 0131, 2323 3375, 2323 9402 *Website* : www.bis.org.in

Regional Offices :

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Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	2323 7617 2323 3841
Eastern	: 1/14 C. I. T. Scheme VII M, V. I. P. Road, Kankurgachi KOLKATA 700 054	2337 8499, 2337 8561 2337 8626, 2337 9120
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